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Recent advances in Catalysis Research using Electron Microscopy

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Electron microscopy provides a highly versatile platform for the characterization of supported metal nanoparticles for heterogeneous catalysis. With both high spatial resolution as well as spectroscopic capabilities, the EM platform can characterize materials in detail. Recent developments include high solid angle EDX detectors, which can rapidly acquire high-resolution elemental maps, and micro electro-mechanical systems (MEMS) based heating holders that can heat samples at very high rates with only little spatial drift. With the addition of environmental capabilities, the microscope can even probe samples under reactive environments.

A recent trend in catalysis is the use of materials that have been engineered at an atomic level. In particular, Density Functional Theory (DFT) is used to computationally screen for new materials. These are often multi-metal alloys, which add new functionality and can reduce the amount of precious metals. Such samples can be size selectively produced either by physical routes, e.g. time of flight mass selection or chemical synthesis e.g. micelle encapsulation. Whereas these approaches may not be technically applicable for large-scale synthesis, they provide a valuable route for gaining fundamental knowledge.

Here, we show findings from three different systems used in three different reactions. Namely, Pt-Y for oxygen electroreduction to H_2O, Pd-Hg for electrochemical synthesis of hydrogen peroxide and ruthenium based catalyst used for methanation [1-3]. With these examples, we illustrate two principle points of nanoparticle functionality: composition and shape.

In the case of the bimetallic catalysts, the elemental distribution in the nanoparticles is of fundamental interest: Do they form a core-shell system or do form an evenly distributed mixture/alloy? Using a high solid angle EDX detector, elemental maps can be efficiently collected and the elemental distribution monitored. Such verification is essential to understand the working principle of the catalyst.

Ruthenium nanoclusters can be used for methanation of carbon monoxide, a reaction used to clean up feed gas for e.g. proton exchange fuel cells (PEM). As-synthesized, the Ru particles assumed high surface-area raspberry-like shapes. However, after treatment under conditions relevant for the methanation reaction, the particles adopted more spherical shapes.

[1] F. Masini *et al.* J. Catal. 308 (2013) 282

[2] S. Siahrostami *et al.* Nature Materials 12 (2013) 1137

[3] A. Verdaguer-Casadevall *et al.* Nano Letters 14 (2014) 1603